IEA SOLAR HEATING AND COOLING PROGRAM

TASK 18

ADVANCED GLAZINGS

and

ASSOCIATED MATERIALS FOR SOLAR AND BUILDING APPLICATIONS

PROJECT B3: CHROMOGENIC GLAZING

FINAL PROJECT REPORT

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1. PROJECT OBJECTIVES

The objectives of Project B3 of IEA Task 18 are to evaluate and compare the properties of a variety of chromogenic glazing, provide the data needed to calculate their energy performance, and determine their suitability for building and solar applications.

2. INTRODUCTION

A chromogenic material changes its optical properties in response to an applied electrical field, ion current, light intensity, temperature, or other stimulus. When this change is sufficient in magnitude and occurs primarily in the visble, solar, or thermal infrared range, then the material may be useful for building and solar applications. Emerging chromogenic glazing technologies, sometimes called "switchable" or smart windows, will forever alter and expand the function of glazing.

Advances in materials science have now reached the point where chromogenic glazing is viewed as a commercial possibility in the next few years. The transition to commercial products, however, will require a large investment in engineering development and fabrication facilities. This investment will only be made if manufacturers see a profitable equation linking device properties, window performance, production cost and sales price. The importance of this project follows from the need to identify the best technologies, to measure and calculate the appropriate performance parameters, and to provide the needed data in useful form to simulators of building performance.

This report summarizes the investigations of various chromogenic technologies by eleven countries: Australia, Canada, France, Germany, Italy, Japan, Norway, Slovenia, Sweden, the United Kingdom, and the United States. A full list of participants and publications are given in the Appendices. First, the current state of research and development was surveyed. Materials and devices were chosen for study based on both promise and availability. Five companies provided samples of electrochromic, thermotropic, and liquid crystal glazing: Asahi, Gentex, Interpane/BASF, OCLI and 3M. The electrochromic materials study covered a wide range of optical, electrical and other physical properties. The device studies focused on the primary optical and electrical or thermal performance parameters. Information determined in this Project was used to provide data and guidance on control strategies for building simulation by Project A2/A3.

3. STATE OF THE TECHNOLOGY

The initial stage of this project was to survey the current activity in worldwide chromogenic research and development. Information was gathered from a wide range of sources and the results were summarized in 1994. [WD1] In the last few years there has been growing interest in chromogenic glazing technology. The potential application of chromogenic glazing is large. The world-wide production of flat glass is about 1 billion m^2 per year. In the U.S. alone, glass production is about 470 million m^2 per year (1988), with about 25% for buildings and 11% for automobiles. Chromogenic products already in the marketplace are rear-view mirrors which can automatically regulate glare in response to incident light levels. Prototype glazing panels are being tested for automobile sun-roofs and visors. Near-future applications include automobile side and rear windows, architectural glazing, aircraft windows and eyeglasses. Chromogenic devices can also be used for large-area information displays in applications where high switching speed is not required, such as for airport display boards. One of the most significant issues is the cost of these devices and the trade-off between cost, benefit, and lifetime. The electrochromic window cost has been estimated to range from 100-1000 US\$/m^2 (10-100 US\$/ ft^2). Some

companies have set cost goals of 100-250 US\$/m². Current research is aimed at long-life devices with durability similar to regular coated windows. A different approach is the development of low-cost limited-life switching devices.

The physical phenomena of interest for optical switching processes can be classified in one of two categories: electrically activated or non-electrically activated as shown in Table 1.

Table 1. Chromogenic Materials

Electrically activated chromogenic materials:

> Electrochromic
>
> Liquid Crystals
>
>> Polymer Dispersed (PDLC)
>>
>> Guest-Host Type
>
> Dispersed Particle Devices (electrophoretic)
>
> Reversible Electrodeposition

Non-electrically activated chromogenic materials:

> Photochromic (responds to light intensity)
>
> Thermotropic (responds to temperature)

The electrically-activated category has the advantage of user control and the non-activated category has the advantage of being self-regulating. Another way that the optical switching phenomena can be classified is into the categories of discrete mass movement or collective physical movement. Discrete mass movement includes ion and localized electron motion as seen in photorefractive, photochromic, electrochromic, and thermotropic materials. Collective physical movement includes thermotropic dispersed and homogeneous liquid crystals, and suspended particles. In Table 2, we list the institutions currently engaged in research and development on electrochromic materials and devices and in Table **3** we list the institutions investigating other types of chromogenic devices. The source of these names is from published papers, patents, trade articles and private communications.

Table 2. Electrochromic R&D Organizations

North America:

Donnelly Corp. (USA)	Dow Chemical (USA)
EIC Laboratories (USA)	Enermodal Engineering Ltd. (Canada)
Ford Glass (USA)	GM Research Labs (USA)
Gentex	Institute National de la Research Scientifique (Canada)
LBNL (USA)	NREL (USA)
Naval Weapons Center (USA)	OCLI (USA)
PPG (USA)	SAGE (USA)
Tufts University (USA)	University of Guelph (Canada)

University of Moncton (Canada)	3M (USA)

South America:

University of Campinas (Brazil)	University Nacional de Eng. (Peru)

Europe:

Bosch (Germany)	CRF-Fiat (Italy)
CTSB (France)	Chalmers Institute of Technology (Sweden)
Conphoebus (Italy)	Dornier (Germany)
ENEA (Italy)	Elf Atochem (France)
Flachglas (Germany)	Fraunhofer Inst. (Germany)
Imperial College (UK)	Latvian State Univ. (Latvia)
Leybold-Heraeus (Germany)	Oxford Brookes Univ. (UK)
P. and M. Curie Univ. (France)	Pilkington (UK)
SIV (Italy)	Schott Glass (Germany)
St. Gobain Glass (France)	University of Bordeaux (France)
University of Catania (Italy)	University of Lanquedoc (France)
University of Rome (Italy)	

Japan & Asia:

Asahi Glass	Canon-Cameras
Carmate-Cars	Central Glass/Nissan Motors (Japan)
GIRI-Nagoya/Sapporo (Japan)	Hitachi-Maxell-Electronics (Japan)
Ichikoh-Cars	Matsushita-Electronics
Murakami Kaimeido-Cars	Nikon-Cars/Eyeglasses
Nippon Sheet Glass	Sanyo Electronics
Sharp-Electronics	Shizuoka University
Takai-Rika-Cars	Tokyo Metro University (Japan)
Tokyo University (Japan)	Topan Printing-Electronics
Toyota Cars (Japan)	Nagoya University

+47 Univ./Co.- Japan, 3-China, 1-Vietnam

Australia:

Glassform	Queensland University of Technology
Sustainable Technologies	University of Technology Sydney

Table 3. Organizations Researching Chromogenic Devices, (Excluding Electrochromics)

North America:

American Optical-Photochromic	Corning-Photochromic Glass (US)

Plastic (US)

GM Research Labs-PDLC (US)	Kent State Univ.-PDLC (US)
PPG-Photochromic Plastic (US)	Research Frontiers-DPS (US)
SunTek-Thermochromic Polymers	Taliq-PDLC (US)

Europe:

Asulab-Liquid Crystals-(Switzerland)	BASF (Germany)
Fraunhofer Inst. Building Physics- (Germany)	Fraunhofer Inst. Solar Energy Systems- (Germany)
Gloverbel -DPS (Belgium)	Interpane-Thermotropic (Germany)
Isoclima-PDLC (Italy)	Pilkington (UK)
Schott Glass-Photochromic Glass (Germany)	St. Gobain Glass-PDLC (France)
Thermotropic (Germany)	Thermotropic polymers (Germany)

Japan & Asia:

Asahi Glass-PCLC (Japan)	Nippon Sheet Glass- PDLC, DPS (Japan)
Nippon Sheet Steel-DPS (Japan)	

4.   ELECTROCHROMIC MATERIALS

The purpose of this work was to determine the effect of microstructure and chemistry on the electrical and optical properties of tungsten oxide electrodes. A variety of studies performed in Canada, France, Sweden, and the UK was summarized [WD2] and then further condensed for this report. Three different types of tungsten oxide were prepared by Optical Coating Laboratory (OCLI) in Santa Rosa, CA, USA. Nominally identical sets of these samples, labeled A, B and C, were distributed to the participants. This study uses procedures developed under the IEA Task 10.[1]

The surface microstructure of the three OCLI films were investigated using scanning electron microscopy (SEM), cross-sectional transmission electron microscopy, and atomic force microscopy (AFM). [FRAl] The average grain size as measured by AFM for samples A and B was about 20 mm and for sample C was 200 nm. Samples A and B had spherical particles with a broad distribution of size and sample C had more ellipsoidal particle shape with a narrow size distribution.

Chemical analysis was determined by using the ion microprobe technique. [FRAl] The chemical composition of sample A was $WO_{3.0:5}$ for a thickness of 360 nm, sample B $WO_{3.10}$ with a thickness of 290 nm and sample C $WO_{2.84}$ for a thickness of about 300 nm. The films had a range of densities; Sample A was 3.6 g/cc, sample B was 4.1 g/cc and sample C was 4.7 g/cc. [JPN1] The structure of all three films was investigated by thin film x-ray diffraction (XRD). It was found that the films were amorphous with the only diffraction peaks noted corresponding to crystalline ITO. Film C exhibited a single unidentified peak not seen in the other samples. [JP?]

The oblique FTIR reflectance spectra of WO_3 films with an incident angle of 70° were measured. From the FTIR spectra, samples A and B are similar while sample C has a different

shaped 3350 (cm-1) O-H band that may be a composite band. [JPN1] Also sample C has an additional absorption peak centered at 1420 (cm^{-1}); the assignment of which is due to an O-H vibration of the bending mode of W-O-H.[2] Therefore sample C has a different hydrated structure from the other samples.

A three electrode cell was used for electrochemical analysis with a Calomel reference electrode and a platinum coated titanium mesh as the counterelectrode. A scan rate of 20 mV/s and a triangle potential cycle of -0.8 to +0.8V was used. Typical voltammograms are shown in Figure 1 after 4 cycles (film was stable). Samples A and B are similar in their voltammetry. However, sample C shows a shoulder on its oxidation peak. This may indicate a minor constituent phase of another form of tungsten oxide. During the first few cycles a certain amount of irreversible charge is incorporated in the film. In sample A, the average incorporated charge is 0.92 mC/cm2 compared to 1.3mC/cm2 for sample C.

Figure 1. Cyclic voltammetry of tungsten oxide/ITO/Glass electrodes in O.1M H_2SO_4, 20 mV/s sweep rate. {UK1?}

Sample A exhibits a high degree of visible and solar transmission modulation. The optical properties vary quickly when inserting charge up a maximum of about 15mC/cm^2 where it saturates. Further insertion of charge up to 43mC/cm^2 does not make any major change in the optical properties. Extracting charge is a much more continuous process. The optical properties vary gradually over the entire charge density range. Sample B exhibits a good degree of visible and solar transmittance modulation. The coloration efficiency (CE) is not as good as sample A. The optical properties variation when inserting charge is not as sharp as sample A and reaches a maximum at 28 mC/cm^2. This sample can store up to 54 mC/cm^2 of charge without any change in optical properties. Extraction of charge in sample B seems to not be a very continuous process. This is due to a certain quantity of charge that is irreversibly inserted in the film B. Sample C exhibits slightly better degree of modulation than sample B. The transmission varies continuously up to about 28mC/cm^2 inserted charge, when it saturates. Further charge can be inserted up to 48mC/cm^2 without any optical change. Contrary to sample A and B, the extraction process shows 2 steps: the optical properties vary suddenly to reach a plateau and then vary again at the end of the range.

Table 4. Summary of all optical properties for samples A-C cycled in 0.1 m H_2SO_4 at a sweep rate of 20 mV/s.

SAMPLE	TRANSMITTANCE		REFLECTANCE		ABSORPTANCE		Total charge	Coloration efficiency
	Bleached Visible/Solar	Colored Visible/Solar	Bleached Visible/Solar	Colored Visible/Solar	Bleached Visible/Solar	Colored Visible/Solar	(mC/cm^2)	(cm^2/C) Visible/Solar
A	0.78 / 0.59	0.05 / 0.04	0.17 / 0.29	0.09 / 0.16	0.05 / 0.12	0.86 / 0.80	42.6	28.0 / 27.5
B	0.67 / 0.55	0.14 / 0.11	0.16 / 0.27	0.09 / 0.16	0.17 / 0.18	0.77 / 0.73	53.4	12.8 / 13.1
C	0.75 / 0.54	0.17 / 0.11	0.12 / 0.24	0.06 / 0.13	0.13 / 0.22	0.77 / 0.76	47.6	13.5 / 14.5

Several investigators used dry and nearly dry lithium electrolytes to evaluate the properties of samples A-C. The electrodes in 1.0 M $LiClO_4$ +PC were cycled using a Calomel reference electrode. [CAN 1] The voltages used were from -1 to +1V. The electrodes were switched in 200 mV steps. Over the range of voltages, sample A switched from 81% to 10% in the photopic spectrum and in the solar spectrum from 71% to 9%. The optical density change was

linear to OD=1.O, with 15 mC/cm^2 injected charge, after which saturation occurred. For sample B the photopic transmittance changed from 77% to 18% and solar changed from 69% to 16%. The optical density changed slope at OD=0.6, with charge of 7mC/cm^2 and leveled off at OD=0.7, with 22mC/cm^2 injected charge. Sample C switched in the photopic spectrum from 87% to 23% and in the solar region from 75% to 16%. Sample C showed an optical density change of OD=O.75 with 10mC/cm^2 injected charge. At higher injected charge levels the optical density actually decreased with OD=O.7, for 20 mC/cm^2 injected charge. This condition is caused by the formation of Li_2WO_4.[3] The best performing electrode was sample A, however, Japan measured identical results for all three samples [JPN1]. They cycled the samples in 1.0 M $LiClO_4$ + PC with 0.1% water by weight using a Calomel reference electrode. The sweep rate was 50 mV/s over a range -1 to 1.2V. Also, chronopotentiometry was performed on the samples and the results were very similar for all the samples.

The samples were cycled in 1.0 M LiCIO4 +PC and found to be fairly stable and they all behaved in a similar fashion. [SW1] A glovebox was used for the cycling tests with lithium foil as a counter and reference electrode. The samples were cycled from 2 to 4 V/Li at 20 mV/s. The transmission properties of the samples are shown in Figure 2. The optical measurements were performed ex-situ. The electrodes were cycled under similar conditions with the potential applied in 0.1 V steps in 60s intervals. In this experiment, however, sample A cracked and fell off the substrate like dust after cycling. [SWE2]

Figure 2. Transmission characteristics of tungsten oxide/ITO/glass electrodes (samples A and C) cycled in IM LiClO4+PC. Both the bleached and colored states are shown. [SWE2]

The diffuse and total reflectance (R_d and R_t and transmittance (T_d and T_t) was measured [SWE2] with a total integrating scattering TIS instrument[4] for films B and C, in the as-deposited, colored, and bleached states. Figure 3 shows the transmittance ratios only. Sample B has a T_d/T_t ratio of less than 0.01 and a R_d/R_t ratio of less than 0.03, irrespective of the electrochromic absorption level. Due to the sensitivity of the human eye, the level 0.01-0.03 is low enough for window applications. Sample C scatters much more in all states and has a T_d/T_t ratio of as much as 0.26 and a R_d/R_t ratio as high as 0.56 for short wavelengths. For sample B there seems to be a tendency of increasing scattering with the number of electrochemical treatments especially, at short wavelengths. This tendency is not seen for sample C. Since the TIS is proportional to the square of the RMS roughness the sample may be getting rougher with cycling. This implies that an increase in TIS with a factor of two as for sample B corresponds to an increase of roughness of approximately 1.4.

Figure 3. Ratio of diffuse to total transmittance and reflectance for samples B and C in the as-deposited, colored and bleached states. The colored state was obtained with inserted charge of 42 and 54 mC/cm2 for samples B and C.

It can be concluded that the Samples A and B are similar structurally and electrochemically. Sample A performed best in both proton and lithium environments, however in one investigation the film flecked off sample A. All samples show a linear region in their charge capacity. The speed of switching for the 3 films is about the same. For samples A and B the ratio between diffuse and total optical response was < 1% and this is low enough for technical applications. Sample C shows ratios of 26% and 56% for the transmittance and reflectance respectively. This scattering is very high and is seen by visible inspection. Film B shows a tendency of increased scattering with the number of electrochemical treatments. FTIR reflectance analysis also shows

that sample C has a unique O-H vibrational mode. Despite the rough microstructure of Sample C compared to sample A the electrochromic performance of sample C was inferior and furthermore showed unacceptable optical scattering.

5.   SMALL CHROMOGENIC DEVICES

5.1.   ORGANIC ELECTROCHROMIC DEVICES (GENTEX)

Samples of electrochromic windows devices from Gentex Corporation of Zeeland, Michigan, USA were circulated for testing in a number of laboratories (Canada, France, Japan, UK, and USA). Results were summarized, [WD3] and one year later, results from long-term cycling of the device were reported. [FRA3] Also, optical scattering and angle-dependent properties were later measured. [SWE4]

The Gentex device tested is a sandwich type system incorporating a liquid phase organic solution (viologen) between two transparent conductor coated glass plates having an approximate active area of 6 cm by 6 cm. All of the devices were initially transparent with a peak transmittance at 560 nm. The original normal transmittance of device #4 was 10% lower than those recorded by other participants, however, and a change in appearance over a period of 5 months was reported. [UK2] In contrast, no change of transmittance, appearance or speed was reported in the aging tests of another sample even after 5000 cycles (Figure 4). [FRA3]

Figure 4. Speed of coloration and decoloration at 497 nm.

To change the device optical properties a DC voltage of up to 1 V can be applied to the transparent conductors. Saturation of transmittance in the colored state was obtained after more than 2 minutes of potential application. The threshold voltage for coloration was about 0.25 V and the most important transmittance change was observed in the visible region. After the wavelength of 1200nm, there was practically no variation in the optical properties with the applied voltage. Figure 5 gives the normal transmittance spectra at different applied voltage.

Figure 5. Normal-transmittance of two Gentex devices: a) Bleached state [CAN2]; b) Bleached state [WD3]; c) Colored state [CAN2]; d) Colored state [WD3].

The average visible and solar optical properties were calculated from the measured normal hemispherical transmission and reflection **Table 5**. [FRA2] There was several percent variation in the measured properties among the participants apparently due to some sample variation and differences in measurement procedure. The absorptance of the devices show that the switch is primarily by absorption rather than by reflection. [JPN2] It was observed that no further variation in the optical properties could be induced when the applied voltage was increased beyond 0.8 V. The response time measurements varied with the applied voltage. [FRA2, CAN2] As a rule, more than 1 minute is needed for stabilizing the coloration but Japan suggested 2 minutes for all cases. [JPN2] The optical memory of the devices is limited to around a minute, necessitating the application of a steady potential for their continuous operation.

Table 5. Visible and solar transmittances, reflectances and absorptances of a Gentex device [FRA2]

Applied Voltage	τ_{vis}	τ_{solar}	ρ_{vis}	ρ_{solar}	α_{vis}	α_{solar}
0.00	76.9	59.8	11.1	11.1	12.0	29.1

0.10	76.9	59.8	11.1	11.2	12.0	29.1
0.20	76.7	59.6	11.2	11.2	12.1	29.2
0.25	76.8	59.7	11.1	11.1	12.1	29.2
0.30	76.3	59.5	11.1	11.1	12.6	29.4
0.35	74.4	58.7	10.9	11.0	14.4	30.2
0.40	66.2	54.8	10.4	10.8	23.5	34.5
0.45	61.4	52.5	9.3	10.3	29.4	37.2
0.50	47.1	46.1	8.4	9.9	44.5	44.0
0.55	32.9	39.5	7.3	9.4	59.8	51.1
0.60	26.7	36.5	6.9	9.2	66.4	54.4
0.70	11.8	28.2	6.4	8.8	81.8	63.0
0.75	8.6	26.1	6.3	8.7	85.1	65.2
0.80	6.9	24.8	6.3	8.7	86.8	66.5
0.85	6.2	24.3	6.3	8.6	87.5	67.1
0.90	5.9	23.9	6.3	8.7	87.9	67.5
0.95	5.9	24.0	6.3	8.6	87.8	67.4
1.00	5.9	24.0	6.3	8.6	87.8	67.4

Color measurements in various states are summarized in Table 6. The calculation of the chromaticity coordinates were based on a type C light source and a 2 degree field of vision. [JPN2] The device color changed from a neutral appearance to bluish green upon coloration. The sample transmittance was measured to calculate the chromaticity coordinates (xy) in comparison with a reference light (D65). Eight test colors were then used to determine the color rendering index which represented the capacity to reproduce the color of the object in comparison with the standard used. The color rendering index was calculated for transmittance as well as reflectance in both the bleached and colored states. [FRA2]

Table 6. **Chromaticity coordinates (x,y) of the colored device based on transmittance spectra [JPN2]**

Mode	Potential (v)	x	y
Bleached	0.0 - 0.25	.3189	.3321
Colored	.55	.2963	.3327
	.65	.2564	.3358
	.75	.2137	.3461
	.85	.1817	.3602

5.2.   INORGANIC ELECTROCHROMIC DEVICES (ASAHI)

Prototypes of Asahi electrochromic windows were distributed to six laboratories including Australia, Canada, France, Germany, Sweden and United Kingdom for optical and electrochemical characterization. The individual reports were summarized in [CAN5]. All laboratories carried out measurements with small samples (5 cm x 5 cm) and France and Germany also gave results for the large windows (30 cm x 30 cm). The layers constituting the windows are: Glass/ TC (200 nm)/ NiO_x (500 nm)/ Ta_2O_5 (500 nm)/WO3 (500 nm)/TC (500 nm)/ adhesive film (250 ^{m}m)/ glass. TC stands for for transparent conductor

Normal-normal transmittance was measured by different participants and a typical result is given in Figure 6. [AUS2] The visible transmittance for ASAHI sample 4 was found to vary from $T_{v,c}$= 8.7 % in the colored state to $T_{v,b}$= 70.5% in the bleached state. The fully colored state corresponded to the state obtained after an injection of 15 mC/cm2 into the device, requiring the device to be held at 1.5V bias for about 10 minutes. The bleached state was obtained by applying a fixed bleaching potential of 1.5V until the current was less than 2 ^{m}A The solar transmittance (AM1.5 spectrum) was found to vary from $T_{s,c}$= 5.2% to $T_{s,b}$= 52.6%.

Figure 6. Normal-normal transmittance of the ASAHI sample 4 in the bleached state and for 3 colored states, with 5mC/cm2, 10mC/cm2 and 15mC/cm2 injected into the WO_3 layer at a constant potential of 1.5V [AUS2].

Corresponding values found for the visible and solar transmittances by the participants are listed in Table 7, but it should be noted that the definition of a fully colored state might vary and hemispherical transmittances were given in many cases instead of normal-normal transmittances. It is evident that for large samples, coloration times were longer. For Germany, for instance, the colored state of a large window was obtained after 30 minutes of applying a colouring voltage of 1.5V. The coloration was generally observed to be quite even in wavelength and spatial distribution on the device surface although others reported regions of different levels of coloration even with a small sample [CAN5, SWE4]. In general however, there was little difference in optical properties between large and small windows.

Table 7. **Visible and solar transmittances (AM1.5, global) in the bleached and colored states for Asahi windows.**

Participant	$T_{v,b}(\%)/T_{v,c}(\%)$	$T_{s,b}(\%)/T_{o,c}(\%)$	Mode
AUS (small)	70.5 / 8.7	52.6 / 5.2	normal-normal T
CAN (small)	71.9 /.4	55 / 5.4	normal-normal T
FRA (small)	69 / 3.7		normal-hemispherical T
FRG (large)	70 / 9	54 / 7	normal-hemispherical T
SWE (small)	72.9 / 6.5		normal-hemispherical T
UK (small)	70 / 8	53 / 5	normal-hemispherical T

Table 8. **Solar and visible transmittances (T), reflectances (r) and absorptances (a) for a small Asahi window [SWE4].**

		T_s	T_v	r_s	r_s	a_s	a_v
Light from	bleached	45.7	72.9	14.9	10.2	39.3	16.9
inside	colored	3.3	6.5	7.9	5.5	88.7	88.0

All laboratories noted very little modulation in the reflectance spectra and it was remarked that the reflectance from the outdoor side was generally higher than from the indoor side, which resulted in lower absorptance.

Figure **7** shows the reflectance from both sides of a small Asahi window.

Figure 7. **Total reflectance spectra for a small Asahi window in both the bleached and colored states. Measurements were made from the *outdoor* and the *indoor* sides of the window [SWE4].**

Detailed measurements of the directional-directional transmittance were carried our for a large Asahi sample. This type of measurements would be quite useful to simulate real situations.

Reductions of transmittance with the angle of incidence both in the bleached and colored states can be seen in Table 9 [FRA6].

Table 9. **Visible and solar directional-directional transmittances for a large Asahi window [FRA6].**

Incidence angle (degree)	Open circuit (T_v%, T_s%)	Applied voltage = 1.5V (T_v%, T_s%)
0	(65.8, 45.9)	(10.2, 5.7)
15	(66.0, 45.8)	(9.4, 5.2)
30	(65.1, 45.8)	(8.9, 4.8)
45	(64.8, 44.8)	(7.7, 4.0)
60	(57.5, 38.7)	(6.2, 3.1)

Measurements of directional-directional transmittance with polarised (s- and p-polarisation) incident light in the interval from 10° to 50° have been carried out by Australia [AUS2] and results have shown little angular variations with p-polarised incident light while substantial changes were observed with s-polarised light. Those results are consistent with preliminary measurements reported by Sweden [SWE4].

Total and diffuse reflectance and transmittance for a small Asahi sample were performed by the Sweden. [SWE4] The total and diffuse transmittances were observed to decrease upon colouring by an order of magnitude. Values for the diffuse transmittance for the bleached state were 2% at about 400 nm and 0.2% at about 1000 nm, and an order of magnitude lower for the coloured state. The same trend in wavelenth dependence was observed for the diffuse reflectance which was consistently lower than 1% for all the spectral region in both coloured and bleached states. Diffuse transmittance was also carried out by Germany for a large Asahi window but the values found were a bit higher than those reported above in the short wavelength range.

CIE-chromaticity coordinates were calculated for a small sample and reported in the paper by Sweden. The Asahi window in the bleached state was quite neutral in colour, i.e. not far from a neutral object characterized by the chromaticity coordinates x=y=z=1/3, with a slightly yellow hue in transmittance. In the coloured state, the window became blueish in both reflectance and transmittance.

Full bleaching or coloration happened in not less than 12 minutes (with an applied fixed potential of +/-1.5V) giving a full cycle of 24 minutes as reported by United Kingdom. [UK3] Others reported a full cycle of 33 minutes [FRA6] or as low as 10 minutes [AUS2] if the colouration time is defined as the time required to reach a transmittance ratio of 8:1. The coloration speed in this latter case was approximatively 400s. Values of 6 minutes for full colouration and 4 minutes for the bleaching process were given by Canada.

To study the speed of colouration and bleaching of a large sample, a spectral multichannel photodetector was used by the Germany. [FRG1] It was observed that after 6 minutes, a reasonable colouration was reached while the bleaching was much faster, the original transmittance being practically reached after 210s. As the electrical conductivity of the Ta_2O_5 layer (ion conductor) is not zero, a slow decoloration will occur even if no voltage is applied to

the window. It was observed that significant bleaching happened after one hour and after a night, total bleaching of the sample was reached.

A small Asahi window was investigated by cyclovoltammetry at a scan rate of 10 mV/s. [FRG1] The cycling was done up to +1.0 V because bleaching occurred at lower voltages than the allowed +1.5 V. No damage was observed after 500 cycles. For the large sample studied by Germany results were not clear due to the long switching time.

The Asahi small and large samples exhibited comparable optical properties which are quite acceptable for applications not requiring high solar transmittance. Angular variations in the directional-directional transmittance, due mostly to the s-component of the incident light, were observed but the changes observed would remain in the acceptable range. Scattering were generally small (less than 2%), although in some cases, it was possible to notice it by eyes. The switching times even for large samples were quite acceptable, as a colouration ratio of 7:1 could be achieved after 7 minutes. The current consumption for the device operation was always fairly low. No serious degradation study with cycling or other factors has been conducted and we could only cite that in principle, a stability of 105 cycles for temperatures between 0°C and 60° C is expected.

5.3.   LIQUID CRYSTAL DEVICES (3M)

Four samples of a glazed, phase-dispersed liquid crystal (PDLC) device, "Privacy Film" manufactured by the 3M company, were distributed for characterization by Austrailia, Canada, France and Sweden and the individual reports were summarized. [FRG4] A PDLC film consists of spherical liquid crystal droplets embedded in a polymer matrix. The typical droplet diameter is about 1μm or less, i.e. of the same order of magnitude as light wavelengths. In a complete PDLC device, the film is sandwiched between two ITO- coated substrates, so that an electric field can be applied across it. On application of a voltage, the liquid crystal molecules are oriented in the field direction, resulting in index matching between the droplets and the matrix, and thus high transmittance, for light propagating parallel to the applied field. When the field is switched off, the liquid crystal molecules reorient randomly, the (unmatched) value of the extraordinary refractive index also becomes effective and causes scattering at the droplet surfaces; the film appears milky.

The samples investigated here consisted of a PDLC device laminated with a polyurethane adhesive between two 2.3 mm thick panes of float glass, resulting in a total thickness of 6 mm. The sample area was 63.5 mm x 70 mm. A maximum voltage of 120 V (AC) is recommended for switching the samples to the transparent state. Glazing of this type is marketed in the USA by Viracon for commercial windows and Marvin Windows for residential windows. Before distribution of the samples, the near-normal hemispherical transmittance was measured with a barium sulfate integrating sphere (diameter 203 mm) in the spectral range from 400 nm to 1800 nm. The values agree within 2.6 % for the on state and 2 % for the off state.

The normal-normal transmittance (T_{nn}) at different voltages in Figure 8 indicates the effectiveness of the PDLC device as a privacy screen, with a near-zero value meaning that images viewed through the sample are obscured. This value does not provide any measure for the effectiveness of the device as a light or energy switch. The most dramatic change occurs on application of the first 30 V, at which voltage Tnnvis and Tnnsol already achieve 75% and 60% respectively of the maximum values. No significant change occurs when the voltage is increased from 100 V to 120 V. The notable transmittance at longer wavelengths even for zero applied voltage indicates that the liquid crystal droplets are too small to cause scattering over the whole spectral range or that the difference in the refractive indices is not large enough. Because of

this, the switching interval for normal-normal transmittance is significantly higher in the visible range (ca. 64%) than for the solar spectrum (ca. 52%). The normal-normal transmittance was found to vary significantly with the polarization angle of the incident and detected light, with the variation also depending on the wavelength of observation. [CAN3]

Figure 8. Normal-normal transmittance spectra for the PDLC device for applied voltages of 0, 10, 20, 30 and 100 V [SWE3].

The normal-hemispherical transmittance is a better measure of the device's efficiency in switching the light or solar energy throughput. The switching range is much smaller than for the normal-normal transmittance; 27% and 19% for the visible and solar ranges respectively. This is mainly due to the high proportion of light which is diffusely transmitted (forward scattered) even when no voltage is applied. Again, this would suggest that the scattering droplets are too small and/or that the mismatch in refractive index is not large enough. It is questionable whether a Tnhsol value of 41% in the scattering state is low enough for the device to prevent overheating in architectural glazing applications.

The spectra in Figure 9 illustrate that the variation in the normal- hemispherical transmittance with wavelength is comparatively slight when determined for non-polarized light and not greatly affected by the applied voltage. Results from other countries show the same relative trends, but there is significant spread in the results not detected as sample difference in the initial characterization.

Figure 9. Normal-hemispherical transmittance spectra for the PDLC device for applied voltages of 0, 10, 20, 30 and 100 V [SWE3].

In real applications, either as a privacy screen or as architectural glazing, the case that the viewer's sightline or the solar radiation is normally incident is the exception rather than the rule. Thus, the dependence of the transmittance on the angle of incidence is an important characteristic. Figure 10 documents the variation in directional-directional transmittance (angle of incidence = angle of transmittance) for applied voltages of 0 and 120 V. In analogy to the normal-normal values, the primary information conveyed here is about image obscuring or haze, not total transmission. However, the already weak dependence on incident angle in the scattering state (0 V) for the directional-directional values indicates that directional-hemispherical visible transmittance would vary even less with the incident angle. Conversely, the pronounced drop in directional- directional transmittance with increasing incidence angle for the clear state suggests that the drop in directional-hemispherical transmittance would be even greater. By contrast, the directional/directional solar transmittance for 4mm float glass is negligibly lower at 30° than at 0° incidence, and even at 60° is still 90% of the value for normal incidence. The detailed angular and polarization dependence of the forward scattering intensity was investigated at a wavelength of 500 nm. [AUS1]

Figure 10. Angular dependence of the directional/directional transmittance spectra for (a) the scattering state (0 V) and (b) clear state (120 V) [FRA4].

As Figure 11 illustrates, there is an increase in the diffuse reflectance when the voltage is increased from 0 V to 100 V. However, the specular component (difference between hemispherical and diffuse) is significantly higher in the clear state due to the reflections at all interfaces, whereas the reflection from the air-film interface away from the light source is negligible for the scattering state. Thus, the difference in the near normal-hemispherical reflectance between the clear and scattering states is considerably lower than for the normal-hemispherical transmittance. The absorptance is higher when no voltage is applied, due to the longer (scattered) light paths within the sample. A solar absorptance of 48% would cause appreciable warming of the sample if it were exposed to solar radiation in a window. Even the absorptance value of 30% for the clear state is significant with respect to solar warming.

Figure 11. Near normal-hemispherical and near normal-diffuse reflectance spectra for applied voltages of 0 V and 100 V [SWE3].

The PDLC sample shows a very high modulation of the normal-normal transmittance with applied voltage but only a 20% increase in normal-hemispherical solar transmittance for the clear state with a high voltage applied as compared to the "opaque" state. The normal-diffuse transmittance and transmittance measurements at variable angles gave a measure for haze, which is considerable at scattering angles greater than 30° even for the "clear" state. These two properties, limited energy modulation and residual haze, suggest that the material is better suited for interior applications as a privacy screen than for architectural glazing.

5.4. THERMOTROPIC DEVICES (INTERPANE/BASF)

Solar overheating in buildings or solar systems can be prevented by windows or panels incorporating thermotropic materials, which change from a transparent to a white, reflective state when their temperature rises, and vice versa. Germany and the UK measured the properties of a thermotropic glazing supplied by Interpane/BASF. [FRG5]

There are two different types of thermotropic materials, hydrogels and polymer blends. At present, the good optical quality of hydrogels filled between two glass panes, combined with their low switching temperatures (20 - 50°C) and rapid switching properties, favor their use for glazing. A hydrogel consists of a relatively mobile polymer component, which is homogeneously mixed with the second component, water in a cross-linked gel, at low temperatures. When the temperature is raised, the two components separate into discrete domains of dimensions similar to light wavelengths, causing scattering of the incident radiation. The size of these domains is determined by chemical modifications of the constituents, which also influence properties like the switching temperature, the switching "gradient" (transmittance vs. temperature) and the switching speed.

A thermotropic laminate consisting of 0.8 mm hydrogel between two 3.9 mm thick float glass substrates (area 100 x 100 mm2) was characterized by measurements of the normal-hemispherical transmittance and reflectance. Angle-dependent measurements of the transmittance and reflectance at a wavelength of 632.8 nm were made to assess the optical quality (minimal light scattering) in the clear state. Further, the sample was submitted to accelerated aging according to the DIN 52344 test for insulating glass units and the change in transmittance determined.

Two different approaches were taken to control the temperature of the sample during the spectral measurements. At Fraunhofer ISE, the sample was mounted in a well-insulated heating

chamber within the standard sample chamber of the spectrophotometer for the duration of a temperature-dependent measurement series. The temperature was regulated by a feedback circuit containing a Pt100 temperature sensor and a hot air blower [FRG3]. At Oxford Brookes University, the sample was brought to the desired temperature in a dry bath, then placed inside an insulating bag with optical ports and transferred to the spectrometer [UK4]. In both cases, the samples were maintained at a given temperature for 20 minutes, to allow them to reach thermal equilibrium, before the spectra were measured.

Figure 12 shows the measured temperature dependence of the normal- hemispherical transmittance spectra of the sample [UK4]. The spectra are relatively flat, apart from the absorption bands due to water in the gel and iron oxide in the glass. This indicates that the discrete domains at raised temperatures are large enough to cause scattering at all relevant wavelengths, not only in the visible range.

Figure 12. Temperature-dependent normal-hemispherical transmittance spectra for 800 μm hydrogel between two 3.9 mm float glass panes: measured spectra [UK4].

Normal-hemispherical transmittance spectra were also calculated for a sample of infinite area using a recently developed Monte Carlo photon-counting program for multiply scattering media.[5] The agreement is satisfactory, apart from the difference in values at 40°C). Near this temperature, where the optical properties change most rapidly, relatively small differences in temperature can result in large differences in the transmittance. Knowledge of the microscopic material characteristics also allows the reflectance spectra for large-area samples to be calculated; the results are shown in.   Comparison of the transmittance and reflectance spectra for a given temperature shows that the sum is less than 100%, the difference being the absorptance in the sample. This increases with increasing temperature and acts to accelerate the switching once it has been initiated.

Figure 13. Temperature-dependent normal-hemispherical reflectance spectra for 800 μm hydrogel between two 3.9 mm float glass panes: calculated spectra for samples of infinite area (derived from measured spectra and corrected for sample/instrument geometry) [FRG3].

The switching gradient of the hydrogel is reasonably steep, as shown in , so that the solar transmittance changes by 33% between 35 and 40°C, as compared to the overall change of 50%. The switching range for the solar values is smaller than for the visible ones due to absorption by iron oxide in the glass and water in the hydrogel.

Figure 14. Integrated visible and solar transmittance and reflectance versus sample temperature.

Angle-dependent optical properties were measured at incident angles of 0 and 60 degrees as a function of the outgoing angle. The most significant result is the narrowness of the transmitted beam indicating that haze is minimal. Further, the ratios of the maximum intensities for the different incidence and polarization angles agree with those expected for a dielectric with a refractive index close to 1.5 in air, i.e. the sample itself does not have a polarizing effect.

The sample was subjected to a constant temperature of 52°C for 14 days, and then daily temperature cycles between +52°C and -15°C for sixteen days and 256 h of UV radiation at sample temperatures between 18 and 28°C, as specified in the German standard, DIN 52344, for IGU's.[6] The switching range in snh,sol (without the geometrical correction) decreased from 55.5% to 52.5%. Slight yellowing decreased the transmittance in the clear state by 1%, and the residual transmittance at 60°C increased by 3%, presumably due to less effective separation of the two components into the microscopic domains. It should be possible to reduce these effects by including a UV filter in the system. The development of small bubbles in the hydrogel around the edge indicates that the edge seal also needs to be improved.

With a temperature-dependent change in solar transmittance between 70% and 20%, the measured thermotropic hydrogel-glass laminate would be an attractive option for variable overheating protection in hot climates. If needed, the transmittance in the opaque state could be easily reduced by increasing the thickness of the hydrogel layer. For temperate climates, the thermotropic laminate can form the outer pane of a heat mirror IGU, which then switches the solar transmittance between 44% and 9%. The product development is at the stage where architecturally sized prototypes have been prepared, but the long-term stability of the materials still needs to be improved before they are released onto the market. The most obvious applications for this type of chromogenic glazing are in overhead glazing in museums, factories and shopping arcades, where the obscured view in the opaque state is acceptable and diffuse lighting is desired. However, it could also be used as vertical glazing near the floor or ceiling level, or as side panels to non-switching picture windows. It thus allows larger areas of glazing overall, and thus enhanced lighting and solar gains when they are needed, without causing overheating in summer.

6.   ENERGY MODELING AND CONTROL STRATEGIES

The Modeling and Control Strategies Project A2/A3 studied the energy and comfort performance of advanced glazing systems including electrochromic and thermotropic technologies.[7] Simulations were performed for the regions relevant to the participating countries and included latitudes from Darwin, Australia at 12 degrees south—a tropical, hot and humid environment, to Sodankyla, Finland at 67 degrees north—with cold winters and mild summers. Finland, Italy, Norway and United States conducted analyses of electrochromic glazings in commercial applications. The United States also studied electrochomic glazings in residential applications and Germany analyzed thermotropic glazings in a residential skylight application. Control strategies affecting both the building envelope and lighting system were studied. Italy performed preliminary work to improve the TRNSYS computer code to facilitate control of electrochromic windows. Table 10 collects the results of the A2/A3 Project on chromogenics only.

Table 10. Summary of Chromogenic Modeling.

Commercial	Application Type	Control Strategy	Energy Savings
Milan (45.75N)	Electrochromic	Temp. and Illum	10-26%
Rome (41.78N)	Electrochromic	Temp. and Illum	10-26%
Helsinki (N)	Electrochromic	Illuminance	25%
Oslo (59.9N)	Electrochromic	Temp. Setback	6-25%
Tromso (69.7N)	Electrochromic	Temp. Setback	4-7%
Blythe	Electrochromic	Illuminance	24-35%
Madison (N)	Electrochromic	Illuminance	no savings

Residential	Application Type	Control Strategy	Energy Savings
Miami	Electrochromic	Illuminance	10% increase
Pheonix	Electrochromic	Illuminance	10% increase
Freiburg	Thermotropic	On/Off Setpoint	10-20%
Bremerhaven	Thermotropic	On/Off Setpoint	10-20%

Results from the chromogenic glazing study are mixed. Commercial buildings at most latitudes show savings from 4 to 26% using either daylight illuminance controls, temperature setback controls, or a hybrid control based on both illuminance and temperature setback. The notable exception is the commercial building in Madison, Wisconsin, USA which showed no savings because of its high cooling load. For residential buildings using electrochromic glazing in Miami and Pheonix, an increase of 10% over static solar control glazing was predicted because there is no savings in lighting energy due to daylighting which is common to commercial buildings. The two thermotropic skylight tests in residential buildings in Freiburg and Bremerhaven, however, both showed energy savings of 10 to 20%.

## 7.	CONCLUSIONS AND FUTURE WORK

This project has surveyed the state of chromogenic technology, attempted to set some standard procedure for testing, compared the properties of several technologies, and helped to demonstrate the potential for energy savings in building applications. The survey shows a rapid increase in the level of interest and commitment to chromogenic research and development. The study of electrochromic tungsten oxide showed that there can be great differences even among materials of the same nominal type caused by differences in growth conditions. A slight level of concern appeared with regard to consistent measurement practice among the laboratories. This information will be used to establish more uniform testing methods for electrochromic materials. Testing of the various devices as expected showed significant differences in detailed properties among the device technologies. This information can be used to provide feedback for device improvements and inputs for building energy performance models. Energy performance studies by Project A2/A3 indicate that in many cases the electrochromic and thermotropic technologies can reduce energy demand by 10-25% compared to conventional glazings. Liquid crystals do not modulate solar heat gain so they would tend to perform like conventional glazing in terms of energy management.

In the few years since the initial survey of chromogenic technology, the level of the technology has changed greatly and prototype products have advanced in size, quality and number. A new study should include several types of large-area electrochromic devices that are now available, as well as interesting new photochromic, photoelectrochromic, and suspended particle technologies. The lessons learned in this project still have to be translated into a standard for optical measurements and a uniform format for simulation of energy performance. This willlead to a basiss for including chromogenic technology into consumer information and government regulatory process alongside of conventional window technology. Optical properties of many technologies, even at the current level, are now generally seen as adequate for energy performance as demonstrated in the current task. Thus, the focus of attention is shifting from energy performance to other factors that will affect introduction of this energy-saving technology to the marketplace such as visual comfort, privacy, durability and cost. A full evaluation of chromogenic technology must include these factors.

## 8.	ACKNOWLEDGEMENTS

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10. APPENDICES

10.1. PROJECT PARTICIPANT LIST

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10.2. NATIONAL CONTRIBUTION WORKING DOCUMENT LIST

T18/B3/WD1/94	International chromogenics glazings technology, a state-of-the-art survey	Lampert, C.M. (1994)
T18/B3/CAN1/94	Reflection, transmission and absorption measurements of OCLI electrochromic samples	Ashrit PV and Truong Vo-Van (1994)
T18/B3/UK1/94	Reflection, transmission and absorption measurements of 3 different types of electrochromic tungsten oxide films	Ageorges P and Hutchins MG (1994)
T18/B3/CAN2/95	Testing of the optical and electrochromic performance of Gentex device	Truong Vo-Van and Ashrit PV (1995)
T18/B3/FRA1/95	Optical and microstructure properties of different electrochromics samples	Chevalier B (1995)
T18/B3/FRA2/95	Optical and electrochromical properties of Gentex device	Chevalier B (1995)
T18/B3/JPN1/95	Optical and electrochemical properties of OCLI WO3 films	Nagai J, McMeeking G and Noutomi Y (1995)
T18/B3/JPN2/95	Optical and electrochemical properties of Gentex device	Nagai J and McMeeking G (1995)
T18/B3/JPN3/95	Characterization of OCLI electrochromic samples	Yoshimura K (1995)
T18/B3/SWE1/95	Electrochemical and Optical Measurements of OCLI WO3 samples A, B, C - 4	Andersson-Faldt AM (1995)
T18/B3/UK2/95	Assessment of the Gentex EC window device no 4 based on a viologen electrolyte	Ageorges P and Hutchins M (1995)
T18/B3/WD2/96	Evaluation of the materials properties of OCLI electrochromic WO3 films	Lampert CM (1996)
T18/B3/WD3/96	Testing of Gentex window devices: summary of results reported by different groups	Truong Vo-Van and Lampert CM (1996)
T18/B3/FRA3/96	Cycling and aging of the Gentex device	
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T18/B3/UK4/96	Optical Characterisation of a Fraunhoffer/BASF Thermotropic Device	Ageorges P, Hutchins MG (1996)
T18/B3/FRA6/96	Optical and Electrochromical Properties of the Two ASHAI Devices	Chevalier, B. (1996)
T18/B3/FRG2/96	Phase-dispersed Liquid Crystal Window (draft)	Wilson, H.R. (1996)
T18/B3/FRG3/96	Optical Characterization of a Thermotropic Device	Wilson, H.R., Nitz, P. (1996)
T18/B3/FRG4/96	Phase-dispersed Liquid Crystal Window (revised FRG2)	Wilson, H.R., Nitz, P.

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T18/B3/CAN3/96	Optical Measurements on 3M PDLS Sample	Ashirt, P.V., Bader, G., Truong, V. (1996)
T18/B3/CAN4/97	Optical and Electrochemical Measurements for a Small Asahi Window Device	Ashirt, P.V., Bader, G., Truong, V. (1997)
T18/B3/CAN5/97	Characterization of Asahi Small and Large Window Prototypes	Ashirt, P.V., Truong, V. (1997)